Close out and Final report for
NASA Glenn Cooperative Agreement NCC3-906

Silicon Carbide Growth

Research

Andrew Trunek has focused on supporting the SiC team through the growth of SiC crystals, making observations and conducting research that meets the collective needs and requirements of the team while fulfilling program commitments. Cancellation of the Ultra Efficient Engine Technology (UEET) program has had a significant negative impact on resources and research goals. This report highlights advancements and achievements made with this cooperative agreement over the past year. NASA Glenn Research Center (GRC) continues to make advances in silicon carbide (SiC) research during the past year. Step free surfaces were used as substrates for the deposition of GaN epilayers that yielded very low dislocation densities. Defect free 3C-SiC was successfully nucleated on step free mesas and test diodes were fabricated. Web growth techniques were used to increase the usable surface area of dislocation free SiC by ≈40%. The greatest advancement has been attained on stepped surfaces of SiC. A metrology standard was developed using high temperature etching techniques titled "Nanometer Step Height Standard". This development culminated in being recognized for a 2004 R&D100 award and the process to produce the steps received a NASA Space Act award.

Aixtron CVD Reactor

Without significant investment in time and research dollars, which would have had a negative impact on meeting program commitments, efforts to attain rotational capability have been suspended. A decision was made to accept and use reactor components that allow SiC research to continue and meet program commitments. This includes the use of a SiC foam insulator and TaC coated graphite components. Although not optimized for yield, the components selected for use are sufficient for proof of concept research currently being conducted. The results obtained to date do not indicate that reactor components are preventing research goals from being obtained. Continued development of reactors and reactor components will be driven by program requirements and resource availability.

3C-SiC By Step-Free Heteroepitaxy

Schottky diodes were fabricated from heteroepitaxially grown 3C-SiC on 4H-SiC substrates. A portion of the epilayer was heavily doped with aluminum. This is suspected of causing a contamination issue with the reactor. Although it was never positively identified as the source of contamination, its use coincided with the presence of the nano contamination. This contamination prevented the realization of atomically flat surfaces suitable for nucleation of defect free 3C-SiC. Each of the nano contaminates would act as nucleation sights for SiC clusters. These numerous nucleation sites would result in a film of highly defective 3C being grown on the surface of the mesa. The realization of defect free 3C-SiC could not be obtained until clean step free surfaces were attained with a repeatable process.

Previously grown defect free 3C-SiC were used for the fabrication of diodes with improved contact metallization. These diodes were successfully operated at high temperatures and under electrical stress with no degradation in SiC crystal structure or failure of the contacts. This indicates that 3C is the thermodynamically stable polytype of SiC while avoiding the localized polytype transformations that have been observed by others [1, 2]. The results of this achievement are scheduled to be presented at the International Conference on Silicon Carbide and Related Materials 20005 (ICSCRM05) by another member of the SiC team.

Step Height Standard or Stepped SiC Surfaces

A significant investment in resources was devoted to development of SiC with highly ordered steps etched into the surface. As technology moves toward the nano scale there is a lack of a metrology standard for calibrating instruments like atomic force microscopes at the nano scale. Taking advantage of the atomic spacing between silicon and carbon atoms and the stacking sequence of hexagonal SiC it is possible to etch SiC to produce steps on the surface of the substrate that have step heights of 0.5 and 1.0 nm (Figure 1). The "Nano Meter Step Height Standard" was selected for a 2004 R&D 100 award. The process for developing the well ordered step patterns on the surface of SiC was titled "Method For Production of Atomic Scale Step Height Reference Specimens With Atomically Flat Surfaces." This process was submitted and selected for a 2004 NASA Space Act award. Currently efforts are underway to transition this technology to the commercial sector.

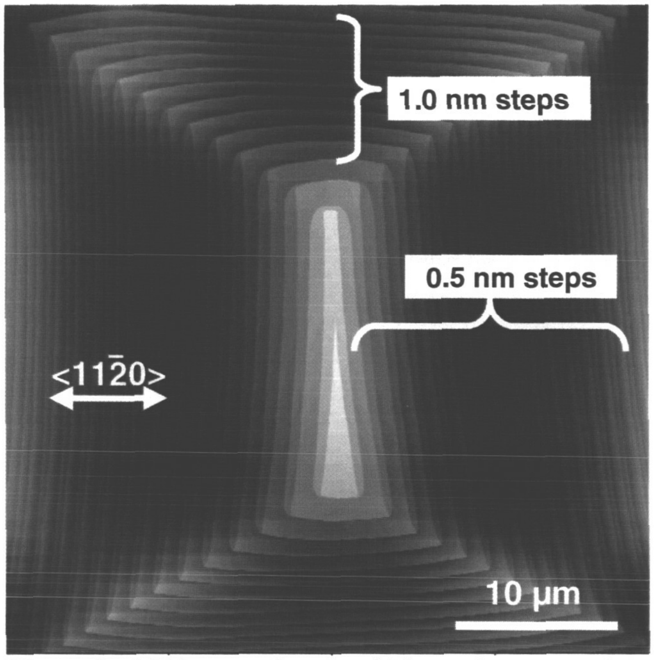

Figure 1. AFM scan of peak of Figure 1 mesa rooftop region showing pyramidal step pattern (rooftop) created by stepflow etching.

Nitrides on Atomically Flat SiC

GRC has continued to collaborate with Naval Research Laboratories (NRL) on the use of step free SiC as a preferred substrate material for the deposition of GaN. Because of the close lattice match between SiC and GaN of $\approx 1\%$ it has been theorized that SiC substrates would yield much lower dislocation densities [3]. However, all previous efforts have used SiC substrates with an intentional off cut angle of 3.5°-8° from the (0001) plane toward <1120> which results in a high density of surface steps. NRL presented results obtained depositing GaN on SIC using GRC prepared stepped and step free SiC surfaces and the associated reduction in defect density. Because of the high density of screw dislocations in commercial substrates on the order of 10^4 cm^{-1} it is impossible to produce large step-free surfaces on as received commercial substrates. This collaborative effort is still in the early stages of development and GRC will continue to provide NRL with step free surfaces for process development.

Expansion of Defect Free SiC by Web Growth

SiC mesas can be expanded latterly through the use of cantilever development. However the limitation on this technique has been unwanted growth in trenches interfering with and stopping cantilever development while inducing dislocations. It has been speculated that if trench growth could be suppressed that cantilever expansion could be continued as long as step flow growth conditions can be maintained. That is maintaining the surface mobility of the adatoms such that they migrate to the edge of the cantilever and incorporate into the crystal structure at the edge. If the adatoms nucleate on the step free surface the stacking sequence information will not be present and the resulting polytype will be 3C-SiC. Development of a selective epitaxial growth mask is being pursued to suppress trench growth. Very recent results using TaC as a mask material indicates that trench growth can be entirely suppressed and the mask will survive extended periods in the extreme environment required for SiC crystal growth (Figure 2).

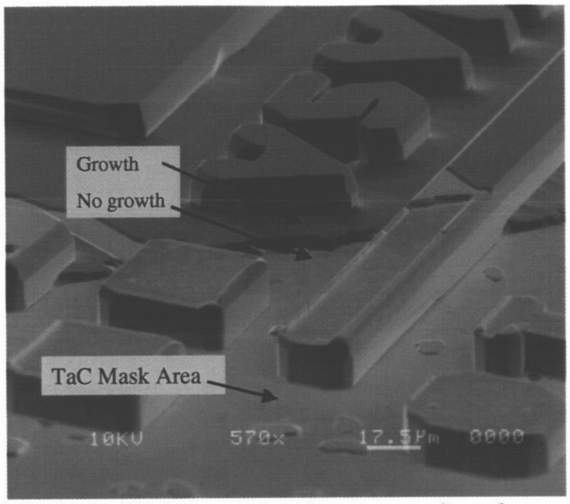

Figure 2. SEM image showing suppression of growth in areas covered by TaC mask.

Gas Sensors

Prototype gas sensors have been fabricated on step free surfaces. This effort has been lead by Dr. Gary Hunter and the results have been very positive [4]. This effort will continue to expand the sensitivity of the sensor by placing the sensing element on a thin cantilever. Current efforts to produce cantilevers sufficiently large enough have been hampered by trench growth. Selective epitaxial growth masks will be employed to realize web based gas sensor technology.

Research Forecast

1. Produce 4H/6H-SiC cantilevers that are completely free of defects on both open and closed mesa shaped patterns with doping profiles suitable for device fabrication. Initial devices would be used for characterizing the quality of the SiC crystal material. This would be followed by large area devices. It is proposed to systematically investigate how dopant atoms are incorporated into the cantilever regions. Achieving the required net donor and acceptor concentrations for device applications will be required.

   Growth in the trench area between mesa shapes has prevented GRC from producing cantilevers on both open and closed mesa shapes of sufficient size for device fabrication. In order to take advantage of GRC's unique mesa growth capability, a selective epitaxial growth mask to suppress trench growth must be developed.

2. Develop Nanometer Step Height Standard parameters for repeatability consistency and maximum yield. This is required for transitioning the technology to the commercial sector.

3. Nucleate and grow the cubic form of SiC (3C-SiC) on step free mesas and cantilevers as a single defect free heteroepitaxial epi layer. The heteroepi would be used for fabrication of SiC devices capable of exploiting the characteristics of 3C-SiC (i.e., MOSFETS and HBT's).

   Large (equivalent to 1 cm^2) areas of 3C-SiC completely free of defects, with sufficient doping, thickness, yield, carrier lifetime, etc. to realize high-voltage power devices in 3C-SiC will be developed. Additionally, small area devices can be realized for material characterization and small signal applications (i.e., sensors).

4. Produce step free regions of increased area to be used as a substrate as part of a collaborative research effort with Navy Research Labs (NRL) for GaN growth on SiC with reduced defect densities. Increase the size of the step free regions using selective epi growth mask techniques.

Technical Papers

1. N.D. Bassim, M.E. Twigg, C.R. Eddy, J.C. Culbertson, M.A. Mastro, R.L. Henry, R.T. Holm, P.G. Neudeck, A.J. Trunek, and J.A. Powell, "Lowered Dislocation Densities In Uniform GaN Layers Grown on Step-Free (0001) 4H-SiC Mesa Surfaces," *Applied Physics Letters*, vol. 86, pp. 021902-3, 2005.

2. D.J. Spry, A.J. Trunek, and P.G. Neudeck, "High Breakdown Field P-Type 3C-SiC Schottky Diodes Grown on Step-Free 4H-SiC Mesas," in *Silicon Carbide and Related Materials 2003*, vol. 457–460, *Materials Science Forum*, R. Madar, J. Camassel, and E. Blanquet, Eds. Switzerland: Trans Tech, pp. 1061–1064, 2004.

3. N.D. Bassim, J.A. Powell, M.E. Twigg, C.R. Eddy, R.L. Henry, R.T. Holm, J.C. Culbertson, P.G. Neudeck, and A.J. Trunek, "Microstructure and Nucleation Behavior of Heteroepitaxial GaN Films Grown on Mesa-Patterned 4H-SiC Substrates," presented at 2004 Electronic Materials Conference, Notre Dame, Indiana, 2004.

References

1. R.S. Okojie, et al., "Observation of 4H-SiC to 3C-SiC Polytypic Transformation During Oxidation," *Applied Physics Letters*, vol. 79, pp. 3056–3058, 2001.

2. R. Okojie, et al., "4H-to 3C-SiC Polytypic Transformation during Oxidation," *Materials Science Forum*, vol. 389–393, pp. 451–454, 2002.

3. S. Einfeldt, Z.J. Reitmeier, and R.F. Davis, "Surface Morphology and Strain of GaN Layers Grown Using 6H-SiC(0 0 0 1) Substrates with Different Buffer Layers," *Journal of Crystal Growth*, vol. 253, pp. 129–141, 2003.

4. G.W. Hunter, P.G. Neudeck, J. Xu, D. Lukco, A. Trunek, M. Artale, P. Lampard, D. Androjna, D. Makel, and B. Ward, "Development of SiC-Based Gas Sensors for Aerospace Applications," *Materials Research Society Symposium Proceedings*, vol. 815, pp. 287–298, 2004.